

2,3-Dibromo-1-(3-bromo-2-thienyl)-3-(4-fluorophenyl)propan-1-one

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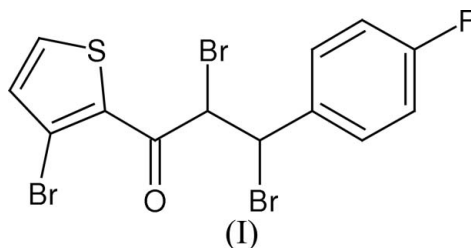
Key indicators

Single-crystal X-ray study
 $T = 173\text{ K}$
Mean $\sigma(\text{C}—\text{C}) = 0.013\text{ Å}$
 R factor = 0.061
 wR factor = 0.154
Data-to-parameter ratio = 14.7For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

Geometric parameters of the title compound, $\text{C}_{13}\text{H}_8\text{Br}_3\text{FOS}$, a chalcone derivative, are in the normal ranges. The bromo substituents at the central C—C single bond are *trans* to each other.

Comment

We have previously reported the crystal structures of (2*E*)-1-(3-bromo-2-thienyl)-3-(4,5-dimethoxy-2-nitrophenyl)prop-2-en-1-one (Yathirajan, Sarojini, Narayana, Ashalatha & Bolte, 2006), (2*E*)-1-(3-bromo-2-thienyl)-3-(2,5-dimethoxyphenyl)prop-2-en-1-one (Yathirajan, Sarojini, Narayana, Bindya & Bolte, 2006), (2*E*)-1-(3-bromo-2-thienyl)-3-(4-chlorophenyl)prop-2-en-1-one (Yathirajan, Ashalatha *et al.*, 2006) and (2*E*)-1-(3-bromo-2-thienyl)-3-(4-methoxy-2,3,6-trimethylphenyl)prop-2-en-1-one (Yathirajan, Narayana *et al.*, 2006). The crystal structure of 2,3-dibromo-1-(2,4-dichlorophenyl)-3-(4,5-dimethoxy-2-nitrophenyl)propan-1-one was reported in the preceding paper (Yathirajan *et al.*, 2007). In continuation of our studies of chalcones and their derivatives, the title chalcone dibromide, (I), was prepared by the bromination of the chalcone, and its crystal structure is reported.



A perspective view of compound (I) is shown in Fig. 1. Bond lengths and angles can be regarded as normal [Cambridge Crystallographic Database, Version 5.28, November 2006, updated January 2007 (Allen, 2002); *Mogul*, Version 1.1 (Bruno *et al.*, 2004)]. The Br substituents at the central C—C single bond are *trans* to each other and the carbonyl group is coplanar with the thienyl ring (Table 1). The two rings are almost coplanar [dihedral angle $5.4(6)^\circ$].

Experimental

(2*E*)-1-(3-Bromo-2-thienyl)-3-(4-fluorophenyl)prop-2-en-1-one (3.11 g, 0.01 mol) was treated with bromine in acetic acid (30%) until the orange colour of the solution persisted. After stirring for half an hour, the contents were poured on to crushed ice. The resulting solid mass was collected by filtration. The compound was dried and recrystallized from ethanol. Crystals of (I) suitable for structure determination were obtained from acetone by slow evaporation

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(yield 80%; m.p. 353–355 K). Analysis for $C_{13}H_8Br_3FOS$: found (calculated): C 33.03 (33.15), H 1.67(1.71), S 6.70% (6.81%)

Crystal data

$C_{13}H_8Br_3FOS$
 $M_r = 470.98$
 Monoclinic, $P2_1/n$
 $a = 11.8008$ (14) Å
 $b = 7.5062$ (8) Å
 $c = 16.758$ (2) Å
 $\beta = 104.010$ (9)°

$V = 1440.3$ (3) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 8.55$ mm⁻¹
 $T = 173$ (2) K
 $0.22 \times 0.20 \times 0.19$ mm

Data collection

Stoe IPDSII two-circle
 diffractometer
 Absorption correction: multi-scan
 (MULABS; Spek, 2003; Blessing,
 1995)
 $T_{\min} = 0.188$, $T_{\max} = 0.203$

6024 measured reflections
 2524 independent reflections
 1826 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.075$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.061$
 $wR(F^2) = 0.154$
 $S = 0.98$
 2524 reflections

172 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 1.74$ e Å⁻³
 $\Delta\rho_{\min} = -1.22$ e Å⁻³

Table 1

Selected torsion angles (°).

Br2—C2—C3—Br3	−178.4 (5)	O1—C1—C11—S1	−2.0 (11)
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H atoms were found in a difference map but were refined using a riding model, with $C_{\text{aromatic}}\text{—H} = 0.95$ Å or $Csp^3\text{—H} = 1.00$ Å, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The highest residual electron density peak is located 0.89 Å from atom S1 and the deepest hole is located 0.85 Å from atom Br1.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in

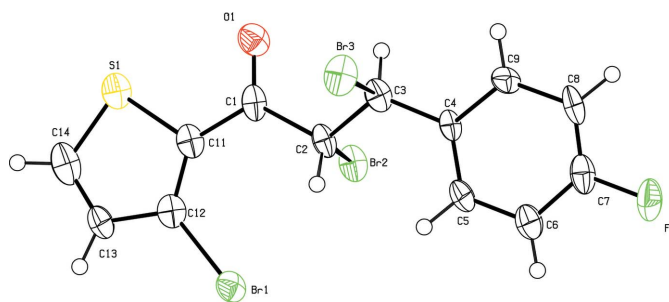


Figure 1

The molecular structure of compound (I), with the atom numbering. Displacement ellipsoids are drawn at the 50% probability level.

SHELXTL-Plus (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97*.

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